

## PATENT SPECIFICATION

779,389



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International Classification:—C07c.

## COMPLETE SPECIFICATION

## A Stable Solid Diazonium Compound and process for making it

We, FARBWERKE HOECHST AKTIENGESELLSCHAFT vormalis Meister Lucius & Brüning, a body corporate recognised under German law, of Frankfurt (M)-Höchst, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

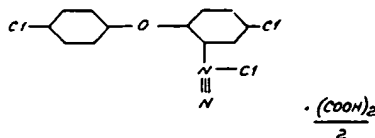
This invention is an improvement in or a modification of the invention forming the subject of Specification No. 20498/54 (Serial No. 778,917).

That Specification describes a process for the manufacture of stable solid diazonium compounds, wherein a diazonium chloride of an amino-azo-compound corresponding to the general formula:



in which R and R<sub>1</sub> represent radicals of the benzene or naphthalene series, is caused to separate out from aqueous solution in the form of a solid oxalic acid addition product by salting out with an alkali metal chloride in the presence of oxalic acid or a water-soluble salt thereof. By this process diazonium compounds are obtained which contain oxalic acid bound in a complex form and which, due to their good stability and solubility, can be used with advantage in dyeing and printing.

The present invention provides the solid, stable diazonium compound which corresponds to the formula



[Price 3s. 6d.]

The invention also provides a process for making the solid stable diazonium compound of the above formula, wherein the diazonium chloride of 2-amino-4:4'-dichloro-diphenyl ether is caused to separate out from aqueous solution in a solid form by salting out with an alkali metal chloride in the presence of oxalic acid or a water-soluble salt thereof.

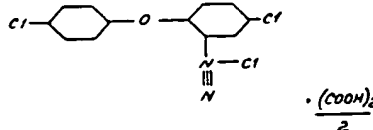
The new diazonium chloride-oxalic acid addition compound is distinguished by its very good capacity to crystallise, and its good stability and solubility. With suitable standardising agents, such as partly dehydrated aluminium sulphate or anhydrous sodium sulphate, it can be worked up to a dyeing salt which can be used with advantage in dyeing and printing.

The following Example illustrates the invention, the parts being by weight:

100 parts of 2-amino-4:4'-dichloro-diphenyl ether are diazotized in a hydrochloric acid solution in the usual manner. The solution of the diazonium chloride is purified with animal charcoal. By slowly adding a solution of 30 parts of crystalline oxalic acid in 150 parts of water and simultaneously cooling to 0—5° C., the diazonium chloride-oxalic acid addition compound separates out in the form of yellowish white crystals. The precipitation is completed by the addition of about 120 parts of sodium chloride. The diazonium compound so obtained may be dried at 45° C. without the addition of a diluent and worked up with a suitable standardizing agent to form a dyeing salt preparation.

What we claim is:—

1. The solid stable diazonium compound which corresponds to the formula



2. A process for the manufacture of a solid stable diazonium compound wherein the diazonium chloride of 2-amino-4:4'-dichlorodiphenyl ether is caused to separate out from aqueous solution in a solid form by salting out with an alkali metal chloride in the presence of oxalic acid or a water-soluble salt thereof.
- 5 3. A process for the manufacture of a solid stable diazonium compound conducted substantially as described in the Example herein.
4. A diazo-salt preparation which contains the solid stable diazonium chloride-oxalic acid addition compound of the formula given in claim 1.
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